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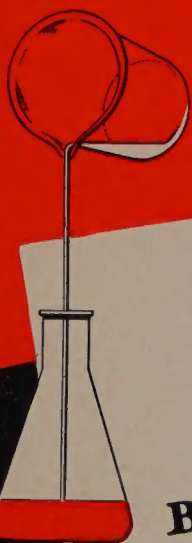


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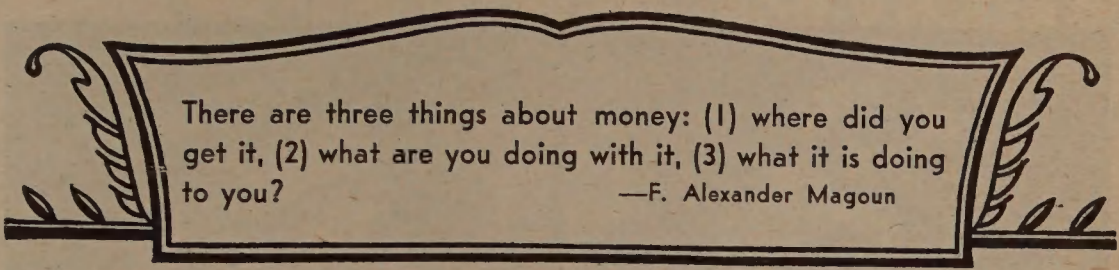
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—F. Alexander Magoun

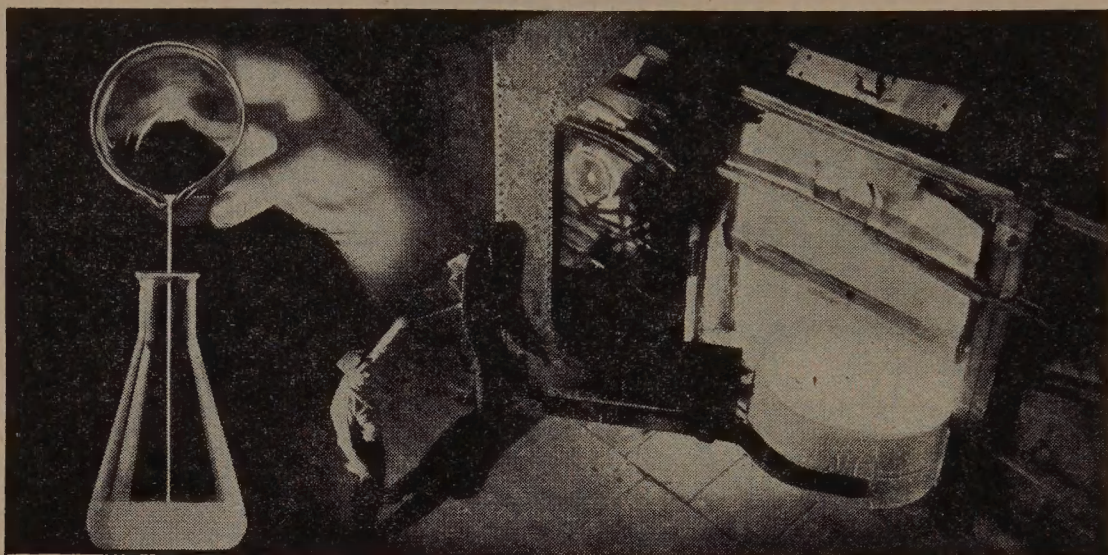
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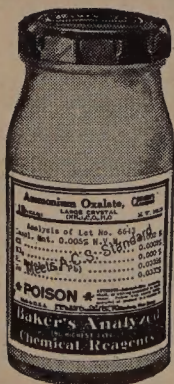
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THE CHEMIST-ANALYST

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S. B. WILDRICK, *Editor*

Recently the Constellation, a new 4-motor TWA cabin plane, crossed the continent in six hours and fifty-eight minutes. California to the East Coast: approximately 2,500 miles apart but only seven hours away.

The significant fact is not that a cabin plane can travel 355 miles an hour, but that the public can see at first-hand what science has made possible.

Hundreds of other scientific accomplishments, just as startling, are largely hidden because of war assignments. Some day their full effect will be felt in business enterprises and in our ways of living. And in most of these miracles of science the chemist has had a part.

Very little has been given the public about the expanding miracles of the electron tube—its application to science, to industry, to recreation. A quiet revolution has been taking place in fibers for textiles. We are beginning to see the widened application of non-ferrous metals, alloys, glass, plastics.

We have heard about synthetic rubber, tubeless tires, 100 octane gas, and unnamed super-fuels, but as civilians we have yet to feel their effects.

Our scientific papers report advanced chemical research in many other lines—soft woods made hard, soils made impervious to moisture, non-melting butter and chocolate which defy temperatures up to 120° Fahrenheit.

These are but a few by-products of our war research program. These and many other developments will create jobs and improve our standards of living for the future. Every chemist should feel proud that chemical research has played such an important role in the war developments, and promises so much for future peace-time pursuits.

BUY MORE WAR BONDS AND STAMPS

Applications of Spot Reactions (IV) *

Note on the Detection of Elementary Sulfur and Selenium

FRITZ FEIGL and NICOLAU BRAILE

Ministério da Agricultura, Laboratorio da Produção Mineral,
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A specific and rapid method for the detection of free sulfur is frequently desired in the testing of materials. For example, it may be found in vulcanized rubber, and many insect powders contain either free sulfur or polysulfides of the alkaline earths. Then some pharmaceutical products contain free sulfur, either as an essential ingredient or formed as the result of an undesirable decomposition. And still another example is the detection of free sulfur in organic solvents, especially motor fuels. Etc., etc.

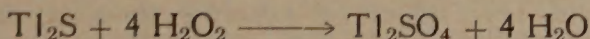
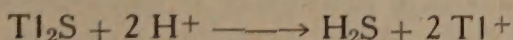
All the usual procedures for the identification of free sulfur in solid products depend on a preliminary extraction of such sulfur with organic solvents, especially carbon disulfide. After evaporation of the solvent a possible residue can then be digested with potassium cyanide or sodium sulfite, and the sulfocyanide or thiosulfate thus formed can be easily detected. A more sensitive test consists in shaking the solution of sulfur with a drop of mercury; even a very small amount of mercuric sulfide is then readily detected by its catalytic acceleration of the iodine-azide reaction (1).

It is frequently overlooked that carbon disulfide has the disadvantage of being able to dissolve only the crystalline form of sulfur. Recently Sommer (2) has noted that this can be avoided by using warm pyridine as the extraction medium. At room temperature, to be sure, pyridine dissolves much less sulfur (about 1%) than carbon disulfide. However, pyridine has the great advantage of being able to dissolve all forms of sulfur, both crystalline and amorphous. At the boiling point of pyridine (114°) the solubility is about 25%, and a blue colloidal solution of sulfur can be produced by boiling the pyridine solution with a saturated solution of NaHCO_3 . This makes possible the detection of 1 γ of sulfur in 1 ml. of solution.

This note will describe a new and easily executed procedure for the detection of elementary sulfur. It presupposes a preliminary extraction. Since the nature of the solvent plays no role, any one of a number of liquids can be used: carbon disulfide, pyridine, ether, etc. The presence of sulfur in a drop of the solution can then be detected by means of a hitherto unknown reaction with finely divided thallium sulfide (3). If a drop of a solution of sulfur (i.e., a solution of sulfur in an organic solvent or in alkali sulfide) is placed on paper impregnated with Tl_2S , the sulfur combines with it to form a reddish brown product, probably $2\text{Tl}_2\text{S} \cdot \text{Tl}_2\text{S}_3$ (3). From the analytical standpoint this polysulfide has the very valuable property of being resistant

* Translation from the German. For the preceding papers, see: THIS JOURNAL, 32, 4, 28, 52 (1943).

to dilute mineral acids and hydrogen peroxide, in contrast to Tl_2S which is dissolved in accordance with the following equations:



The formation of this thallium polysulfide is immediately perceptible when a drop of an ammonium polysulfide solution or of a concentrated solution of sulfur in an organic solvent is placed on paper impregnated with black Tl_2S . After evaporation of the solvent there remains a brownish red fleck which is distinctly visible even in the presence of the black Tl_2S . In the case of dilute solutions of sulfur the formation of the polysulfide is not recognizable directly, but it can be easily detected by its resistance to acids or hydrogen peroxide. It is sufficient to lay the paper in dilute acid or hydrogen peroxide solution; then the uncombined Tl_2S dissolves almost instantly and the black paper becomes white. Only at the spot where the thallium polysulfide could be formed does there remain a brownish red or brown fleck.

In this way even 3 γ of sulfur can be detected in one drop (0.05 ml.) of solution.

Solutions of selenium in organic solvents or in colorless alkali monosulfide behave like solutions of sulfur. The addition product formed by the Tl_2S and selenium possesses a black color and is, therefore, not to be recognized directly on the black Tl_2S -paper. If, however, the paper is placed in dilute acid or hydrogen peroxide, the Tl_2S dissolves and at the spot there remains a dark brown to black fleck. The limit of identification of a test carried out in this way amounts to 1 γ of selenium in the drop.

These tests for the detection of sulfur and selenium by the formation of addition compounds with thallium sulfide cannot be carried out in the form of test-tube reactions but only in the form of spot reactions on paper impregnated with thallium sulfide. This is due to the fact that they utilize two effects which are connected with the surface and capillary actions of the paper. These effects are of quite general importance for spot reactions on paper (4). One is the rapid transposition of the finely divided substances in the capillaries of the paper in consequence of their large free and reactive surface. In the present case it is the formation of thallium polysulfide or polyselenide. This addition of sulfur or selenium is distinctly visible only when solutions of sulfur or selenium act on finely divided Tl_2S in the paper. In a test-tube the addition can be realized either not at all or only very slowly, and probably for this reason it has not hitherto been observed. The second effect on which the sensitiveness of these tests depends is the possibility of a very extensive covering of the surface of a finely divided insoluble substance in the capillaries of the paper with a reaction product produced thereon. By this covering the unchanged portions beneath can be protected from the action of the solvent and the like in so far as the reaction product is resistant to them. In the present case the addition product formed on the surface of the Tl_2S protects the unchanged Tl_2S beneath from solution by acids or hydrogen peroxide. As a consequence of this the color of the protected Tl_2S is added to the color of the thallium polysulfide or polyselenide which forms on the spot. In the

case of very dilute solutions of sulfur or selenium it is probable that the fleck produced on the Tl_2S paper consists almost entirely of unchanged thallium sulfide, protected by amounts of polysulfide or polyselenide which by themselves would be imperceptible. Other experimental results support this conception of a protective layer effect; for example, the great sensitiveness of the spot reaction for palladium on nickel dimethylglyoxime paper undoubtedly depends on the same principle (5).

Reagents

Thallium Sulfide Paper. Filter paper (Carl Schleicher & Schüll No. 589 or Whatman No. 42) is soaked for several minutes in a 0.5% solution of thallium carbonate or acetate. It is then drained and dried with warm air. For the formation of thallium sulfide the paper is placed over the mouth of a beaker containing a solution of ammonium sulfide, warmed to about $80^\circ C$. Within a few minutes the paper is fully blackened and the transposition to Tl_2S is completed. The paper, cut into strips, can be used directly for the experiments.

Freshly prepared reagent paper should always be used. For when it is stored, partial decomposition takes place in consequence of oxidation.

Nitric Acid. A 0.5 N solution.

Procedure

A drop of the solution to be tested is placed on freshly prepared Tl_2S -paper. Either the solvent is allowed to evaporate spontaneously or a current of warm air is blown on the paper. It is then moved to and fro in a little dish containing dilute nitric acid. When sulfur and selenium are absent, the entire paper becomes white in a short time (about 30 seconds). In the presence of sulfur or selenium a brown or black fleck remains on the spot. The intensity of the color of the fleck depends on the amount of sulfur or selenium present. The fleck having been formed, it is recommended to lay the paper in a dish of water in order to prevent further action of the acid and a gradual fading of the fleck.

The intensity of the color of the fleck enables us to tell whether appreciable amounts or mere traces are present in the solution undergoing test. Comparisons are made with solutions of sulfur (or selenium) of known concentration. After thorough washing with water and drying, the spots on the Tl_2S -paper are stable.

Applications

When solid materials such as insecticides or pharmaceutical products are to be tested for elementary sulfur, a finely pulverized sample is treated with carbon disulfide, pyridine or the like in an extraction apparatus. Since only a drop of solution is required, very small amounts of materials and micro-apparatus can be used; this is an advantage in regard to the expenditure of both time and material. Let it be noted that the completion of a sulfur extraction with organic solvents can also be ascertained with one drop of solution by means of the procedure described. This is of use in the quantitative determination of sulfur or in the purification of precipitated sulfides (HgS , MoS_2 , etc.).

In order to detect free sulfur in rubber, it is sufficient to place a small piece of the material in a test-tube and keep it in contact with CS_2 for 2-3 minutes. When a drop of the solution is placed on Ti_2S -paper, there remains a distinct fleck of polysulfide after the treatment with acid.

In sulfide ores, gas purifying materials and the like, free sulfur can be detected in the following manner. A few milligrams of the dried and finely pulverized material are placed on Ti_2S -paper and moistened two or three times with a drop of carbon disulfide. After evaporation of the carbon disulfide the dry powder is brushed off the paper which is then placed in the dilute acid. The formation of a dark fleck proves the presence of free or extractable sulfur. In this way considerable amounts of extractable sulfur could be detected in pyrite, marcasite and galena; in zinc blend only traces were found.

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Weisselberg, K., *Petroleum Z.*, **31**, No. 10, 7 (1935).
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Book Review

Laboratory Manual of Spot Tests. F. Feigl (Translation by R. E. Oesper).
xii + 276 pp. Academic Press, Inc., New York, N. Y., 1943. (\$3.90)

As stated in the Foreword, this book has been written expressly as a text for introduction of spot analysis into the curriculum as an added tool for the use of the busy analyst. As spot analysis itself presupposes more than an elementary knowledge of chemistry and physics, the text is definitely suggested as a laboratory manual and treatise for a special course to acquaint not only the student who may have used some of the tests and techniques as adjuncts in the classical courses in qualitative analysis but also for the practising chemist to whom a saving of time and material may be of utmost importance.

The theoretical foundations for the various techniques employed are thoroughly and understandably covered throughout the text, exemplifying experiments having been judiciously introduced.

Reactions for identification of various substances are covered under generic and specific headings. Thus for example, reactions for detection of inorganic substances are treated under five generic headings, each of which is subdivided into from three to eleven subheadings or species. These genera and species cover practically all the methods of detection which lend themselves to such type reactions.

Qualitative organic analysis by spot tests range from reactions for the detection of elements and groups present in organic molecules to reactions for organic molecules capable of reacting as units.

The methods for the analysis of minerals, trace analysis of metals in water and commercial products and procedures applicable to biological substances should be of interest, as well as the chapter on confined spot tests and spot colorimetry.

The text is very free from errors and is written in an easily understandable style as might be expected from the pens of the author and translator. The experiments are well thought out and lend themselves readily to exemplification of the author's intentions. The reviewer has followed this field closely for the past fifteen years and has only one criticism, a minor one, that a few more of the actual spot tests might have been included although this might make the text too large and cumbersome for use in college courses.

W. A. HYNES

Fordham University, New York City

Determination of Dimethylglyoxime Required to Remove Nickel from Plant Solutions

PAUL VON STEIN
Elyria, Ohio

It was necessary to solve the problem of removing nickel from large plant batches of liquor intended for use in the production of cadmium pigments. And it was decided to employ dimethylglyoxime, as it had been found that this expensive chemical could be recovered by suitable treatment of the nickel dimethylglyoxime precipitate. A control test was required to determine the volume of dimethylglyoxime solution required to precipitate the nickel present in these batches. Since the usual gravimetric procedure was too time consuming, the following volumetric method was developed.

Method. An accurately measured 5 ml. of the dimethylglyoxime solution is placed in a 250 ml. beaker. This is followed by 5 ml. of glacial acetic acid, 50 ml. of water and 10 ml. of concentrated NH_4OH . The resulting solution is heated to 95°C . and titrated with the liquor to be tested. This liquor is added dropwise from a burette and with vigorous stirring. The end point is determined by means of test paper, prepared by impregnating filter paper with a 10% solution of nickel sulfate and subsequently drying. One drop of the solution in the beaker is transferred to a piece of this test paper with a stirring rod. The end point is reached when the solution no longer produces a red coloration of the paper. Care should be taken not to mistake any red flecks coming from the beaker for the end point of the reaction.

A slight excess of the dimethylglyoxime is desirable. In working with 1000 gallon batches of plant liquor it has been the author's experience that in no instance will the excess be greater than one gallon of a 3% solution.

A Simple Extraction Procedure for the Rapid Determination of Carotene in Garden Vegetables

LEONARD P. PEPKOWITZ and JOSEPHINE GARDNER

Department of Agricultural Chemistry
Rhode Island Agricultural Experiment Station
Kingston, R. I.

When using the Waring Blendor for the extraction of carotene from plant tissue, the usual procedure is to extract the samples with 200-250 ml. of extractant, filter the extract into a 500 ml. volumetric flask, wash out the grinding assembly quantitatively and continue until the washings are colorless. This procedure is laborious and time-consuming and not adapted to routine procedures. Moreover, it entails the use of a large amount of acetone per determination, which is a serious consideration at the present time.

The following simple modification was developed to eliminate these objections and to provide a technique applicable to a large number of routine determinations. Its effectiveness has been demonstrated by several hundred determinations performed at this laboratory.

To a 30-50 g. sample of fresh tissue placed in the Waring Blendor, 200 ml. of 95% acetone are added from an accurately graduated cylinder or dispensing burette in order to make the final concentration of acetone in the extract approximately 85%. Smaller samples of dehydrated tissue, 3-5 g., may be extracted with 200 ml. of 85% acetone.

The volume, V , of the solution containing the carotene will then be the sum of 200 ml. and the number of milliliters of water in the sample. The material is extracted for 5 minutes and then decanted through a medium filter paper. A measured portion, v , of the filtrate, dependent on the concentration of the carotene, is taken directly for analysis; this is usually 25 or 50 ml. for the fresh tissue of vegetable crops.

In order to express the concentration of carotene obtained as milligrams of carotene in 100 grams of sample (mg. %), the following expression may be used:

$$\text{mg. \%} = \frac{100 C V}{S v}$$

wherein S is the number of grams in the sample taken and C is the number of milligrams of carotene found in the v milliliters of the solution.

Table I presents the carotene concentration of a number of vegetables determined by the usual procedure and by the proposed modification. The results are in agreement and indicate the relative accuracy of the procedure.



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LABORATORY APPARATUS AND CHEMICALS

TABLE I

Vegetable	Original Procedure	Modification
Spinach, fresh	2.83 mg. %	2.62 mg. %
Pepper, fresh.....	0.73	0.73
Lettuce, fresh.....	2.61	2.74
Kale, fresh.....	5.52	6.00
Spinach, dehydrated	25.9	27.5
Carrots, dehydrated	11.1	11.3
Soybeans, dehydrated.....	0.63	0.60

Diffusion in Solution

S. B. BUTLER

Queens College, Flushing, New York

Most courses in elementary chemistry include a demonstration of diffusion in solution. This is illustrated by placing a quantity of copper sulfate in the bottom of a tall cylinder filled with water. The cylinder is then placed on exhibition so that the class may see the salt slowly distribute itself throughout the liquid. The process is supposed to require months or years to make the solution uniform.

But in actual practice this demonstration is not always effective. Because the solutions of most colored salts do not show conspicuous differences in color once a moderate concentration is passed, the contents of the cylinder appear to be uniform to the uncritical eye of the student after a very short period, a few weeks.

The experiment may be considerably improved by using a salt which yields concentrated solutions of a different color than its dilute solutions. A good example is cupric dichromate. Moderately strong solutions are dark brown, practically opaque, while dilute solutions are pale yellow or orange. Its lower solubility makes it more economical and effective to use than cupric bromide, which yields brown (concentrated) and blue-green (dilute) solutions. Using cupric dichromate in a cylinder 4" x 30", the level of the brown concentrated solution rose only half way in six months.

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Microchemical Methods in Drug Assay

M. P. SCHESTER

Technical Laboratories, Sears, Roebuck & Co., Chicago, Ill.

The manifold advantages of microanalysis, which have led to its adoption in a wide variety of fields both in pure science and industry, led to the investigation of its possibilities in the control of drug products. It was found that its application in the assay of drugs which undergo a periodic check in these laboratories resulted in great economy of the amount of attention required of the analyst, reductions of running time by more than half in all cases, and substantial saving of reagents. Precipitations are easily and quickly made; filtrations and washings require only about five minutes; and drying time is cut down, sometimes by as much as several hours, with no sacrifice of accuracy. Results of microassays have been found to check well with macroanalyses of the same products.

The present work includes the results of trial analyses of only a few products, but these assays may be applied to other preparations containing the same materials and the usefulness of microanalysis may be greatly extended in this field by the investigation of other microassays. The analytical procedures are standard micromethods which may be studied in complete detail in the original papers to which the reader is referred. The purpose of this paper is to describe only the applications and necessary modifications of these procedures when applied to drug assay. The usual precautions in sampling, handling and weighing are to be observed and will not be described here.

Zinc Ointment

Determination of Zinc. A 5-7 mg. sample is weighed out in a small porcelain crucible, and the crucible is placed in a furnace at about 750-800° where the organic matter is quickly burnt off. The crucible is next carried to the electrolytic apparatus on a nickel block. Two drops of 2 N hydrochloric acid are added and the crucible is allowed to stand for 2-3 minutes. When solution of the zinc oxide is complete, the crucible is carefully picked up with platinum shod forceps and its contents are washed quantitatively into the electrolytic cell of Clarke and Hermance (1). An equal volume of 20% KOH is added and the zinc is plated from this 10% KOH solution onto a cathode previously coated with copper as directed by Clarke and Hermance.

Conditions to be carefully observed are: temperature, 25°; E. M. F., 3 volts; time, 15 minutes; concentration of KOH, 10%.

Results: by macromethod, 20.1 %; by micromethod, 20.2 %.

Tablets Containing Epsom Salt and Phenolphthalein

Determination of Magnesium (2). Weigh out about 8 mg. of finely powdered sample into a glass microbeaker, add 1 ml. of water, carefully render acid with dilute HCl (the phenolphthalein acts as indicator), and digest for 10 minutes on the steam bath. Filter through the glass-asbestos filter stick into a porcelain crucible previously weighed with the filter stick and wash with small (0.5 ml.) portions of water. Add several fragments of Na_2HPO_4 to the acid solution which is then heated to boiling. To the hot solution

add enough 10% ammonia to render it alkaline and then an additional 2-3 ml. Allow the precipitate to stand overnight under a glass bell jar. In the morning the mother liquor is drawn off through the filter stick and the precipitate is washed with 2-3 % ammonia. The crucible and filter stick are then placed in a small muffle and ignited, either by starting with a cold muffle and allowing it to come up to a final temperature of 1000° or by previously drying in an oven and placing in the hot muffle.

Results: by macromethod, 359 mg. MgSO_4 per tablet; by micromethod, 361 mg.

Tablets Containing Sulfur and Cream of Tartar

The sulfur in this product is assayed directly by the volumetric modification of the Pregl spiral tube method (3). The economy of time is extremely striking here, about ten times as much time being required for the macroassay as for the microdetermination. For precise directions the reader is referred to the original.

Results: by macromethod, 23.15%; by micromethod, 23.25%.

Alum

Determination of Aluminum. The procedure used is the one described by Benedetti-Pichler (4). Aluminum is precipitated as the oxyquinolate, which is then dried and weighed. This assay can be performed in about one hour of actual working time.

A 7-8 mg. sample is weighed into a microbeaker and dissolved in 1 ml. of water. A drop of HCl is added and then 0.5 ml. of the oxine reagent.* The beaker is placed on a steam bath and 2 N ammonium acetate is added until the first permanent turbidity is observed. After a minute 0.5 ml. more of the ammonium acetate solution is added. The beaker remains on the steam bath for 10 minutes. Then the mother liquor is drawn off through the filter stick and the precipitate is washed four or five times with $\frac{1}{4}$ — $\frac{1}{2}$ ml. of water. The precipitate is dried at 140° in the apparatus of Benedetti-Pichler and weighed in the usual manner.

Results: sought, 11.26% Al_2O_3 ; found, 11.24% (average).

The assays of other materials by these methods will suggest themselves. We hope to continue along these lines in order to utilize the economy afforded by microanalysis in the examination of other drug preparations.

The macroanalytical work of Dr. K. Graham and Mr. C. N. Sprankle in checking the microdeterminations and their many helpful suggestions are gratefully acknowledged.

*Grind 5 g. of 8-hydroxyquinoline with 12 g. of glacial acetic acid and then add 83 g. of water.

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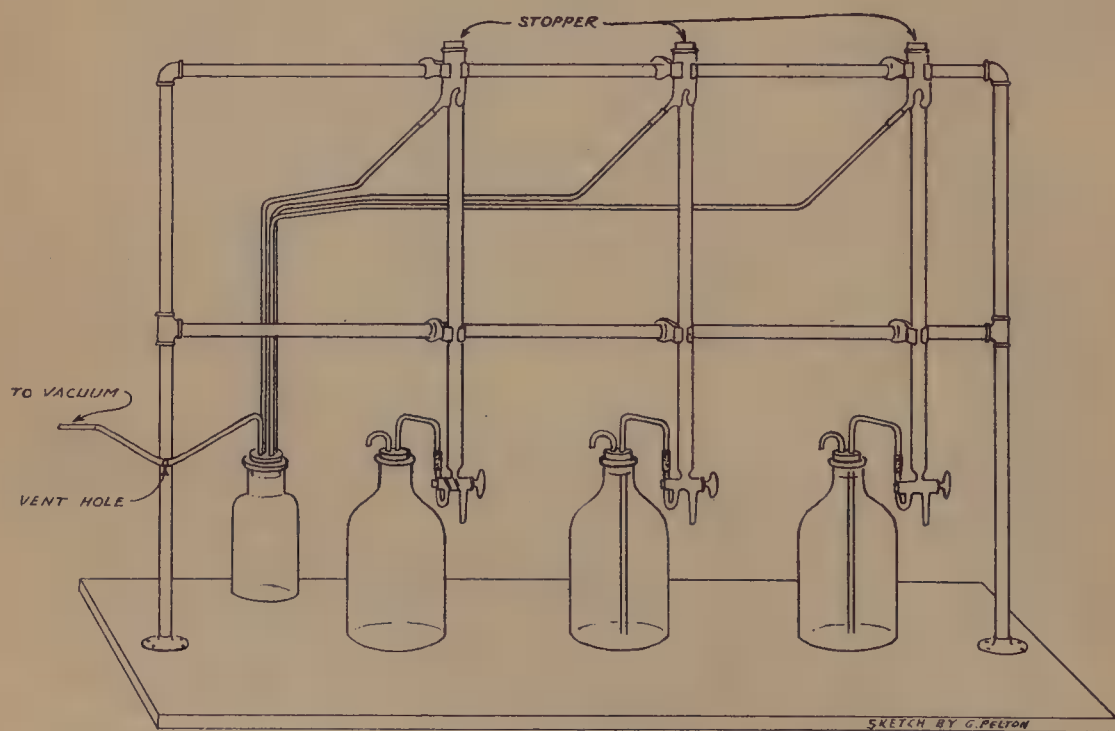
LABORATORY SUGGESTIONS

An Automatic Burette Set-Up

BENJAMIN S. FLUG and RAYMOND E. KARCYNSKI
Grand Rapids Brass Company, Grand Rapids, Mich.

The automatic burette set-up described here has been found to be very satisfactory and a great time saver in our control laboratories. This set-up has a further advantage over some gravity feed systems in that the reagent does not have to be pumped back to the stock bottle.

The filling of the burette is accomplished by means of a vacuum from a pump or an aspirator, and, although all the burettes are hooked up to one vacuum line by means of the trap, each can be worked independently.



After the vacuum line is opened, the burette is filled by holding a finger over the vent hole (see sketch) and turning the stopcock into position to connect with the stock bottle. When the burette is filled, turn the cock and remove the finger from the vent. Any overflow will, of course, run down to the trap. The trap should be arranged in such a way as to allow emptying when necessary.

A Constant Temperature Control

ALLEN E. SMITH
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Constant temperature baths are often needed in the laboratory for physical chemistry experiments and some analytical procedures. Standard laboratory equipment may be used to make a reasonably accurate thermostat. Only the most elementary glass blowing is required and no relay is used.

A long stem thistle tube is bent to form a "U" and one arm of a T-tube is sealed to it, as shown in the accompanying drawings. A smaller tube, *M*, fits snugly into the other arm of the T-tube. A coarse 6'' wire, *W*, is sealed in the lower end of *M*, and a small quantity of mercury in the tube makes it possible to wire the system. A 1'' length of small diameter rubber tubing, *R*, is used to form an air-tight seal with *M*. Mercury is poured into the thistle until the level is such that some of the mercury remains in the thistle, as shown in Fig. 1. The tube, *M*, is placed in such a position that it may be

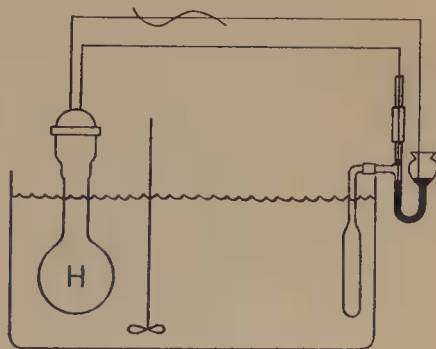
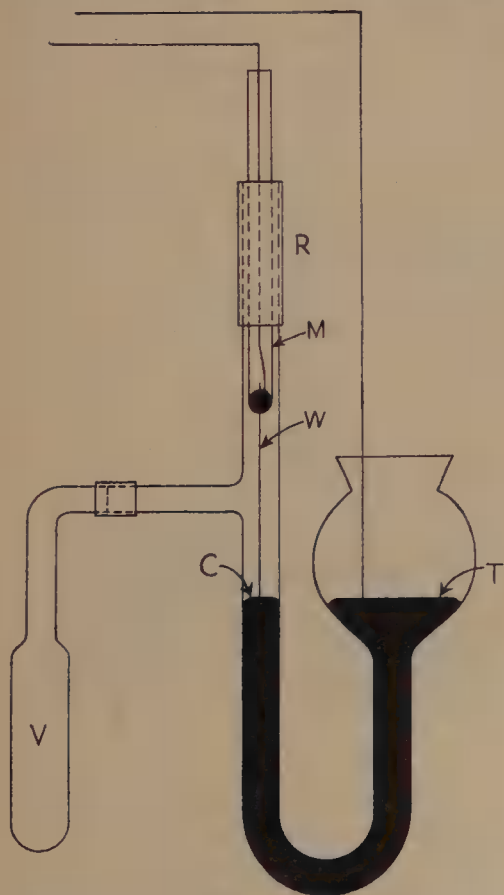


Fig. 2.

raised $\frac{1}{2}$ " or lowered about $\frac{3}{4}$ " by sliding it through the rubber tubing, *R*. The wire is then cut so that it just touches the mercury meniscus at *C*. The stem of the T-tube is provided with a piece of rubber tubing so that an air-tight connection may be made to a vessel, *V*, containing a volatile liquid. The circuit is wired, as shown in Fig. 2, and the apparatus is then ready for use.

If 25° is the temperature to be maintained, CCl_4 is a suitable liquid to place in the vessel. The water in the bath is brought to approximately 1°C. below the desired temperature and the vessel containing the carbon tetrachloride is immersed in this bath. When the CCl_4 has attained the bath temperature, the vessel is connected to the T-tube as illustrated in Fig. 2. The contact at M is adjusted so that it just makes contact with the mercury, causing the light-bulb heater, H , to operate. As the temperature rises, the enclosed gas expands and the vapor pressure of the CCl_4 increases (about 5.2 mm. per 1°C.). This increase of pressure pushes the mercury down at C , breaking contact and shutting off the heating device. Slight further adjustment may be necessary to make the switch open and close at the desired temperature.

The advantage of using a thistle tube instead of plain glass tubing lies in the fact that, if the latter is used, a 5 mm. increase in vapor pressure changes the level of the mercury at C by only $2\frac{1}{2}$ mm., since the level at T rises an equal amount. In this device the sensitivity is doubled, because the mercury level in the thistle is almost unaltered by a 5 mm. depression at C .

A small thin-walled vessel with relatively large surface and small volume should be used. Not a great deal of the volatile liquid is required, as the change in vapor pressure is independent of the amount as long as the liquid phase is present.

For higher temperatures a different liquid may be used, although for high boiling liquids the sensitivity is lowered, since the rate of change of vapor pressure with temperature is smaller. Carbon tetrachloride is satisfactory up to about 60°C. , except for its slight tendency to decompose and form mercury chloride.

The advantages of this thermostat are: ease of construction, low cost and the elimination of electric relay. With suitable liquids and a thin-walled vessel, it is accurate to within 0.2° near room temperature and within 0.8° for a reasonably wide range.

Removal of Grease Plugs from a Burette Tip

JOSEPH J. KOLB

Lankenau Hospital Research Institute, Philadelphia, Pa.

The commonest method for the removal of a grease plug from the tip of a burette is boring the plug out with a fine wire. The next most commonly used method is immersion of the tip in chloroform or carbon tetrachloride. If the plug forms during the course of a titration, the titration is usually ruined. Such a titration may be salvaged by use of the method to be described.

Dry the tip of the burette. Open the stopcock. A lighted match is passed back and forth beneath the tip of the burette, while the other hand is on the stopcock to close it as soon as the plug is gone. One or two passages of the lighted match are usually sufficient.



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Salvaging Broken Thermometers

D. H. MATHESON

Hamilton, Ontario, Canada

Thermometers whose stems have been broken can frequently be salvaged to make very useful instruments covering a more limited temperature range. For example, a broken 360°C . thermometer might make a satisfactory instrument reading from -10 to $+50^{\circ}\text{C}$., which would be about 4 inches long and suitable for carrying in a clinical thermometer case.

Since the mercury thread is apt to be broken and possibly part of the mercury lost, it is necessary to check the reading of the broken instrument against a standard thermometer. If the indication is incorrect or the thread is broken, it will be necessary to refill the capillary. Heat the bulb in a water bath until all of the broken thread is forced out of the open end of the capillary. The mercury will collect in a little globule to which an additional little droplet can be added. On cooling the bulb the mercury will contract and an intact thread of mercury will be drawn into the capillary.

To adjust the length of the thread, note the scale indication where the stem is broken off. Attach the broken thermometer to the standard and place in a water bath heated to exactly this temperature. The excess mercury which is forced out can be wiped off, and on cooling the thread will be withdrawn and will indicate the correct temperature. It is not difficult to adjust the thread accurately to within one quarter of a millimeter in this way.

In sealing the thermometer, a small bulb must be provided at the top of the capillary to accommodate the gases forced out when the mercury thread rises in the tube. Place the bulb of the thermometer in a test-tube of freezing mixture to contract the mercury as far as possible. Heat not more than 4 mm. of the upper end of the stem in the flame and rotate it to allow a smooth round end to form. Then, while this end is still hot in the flame, remove the test-tube of freezing mixture and heat the bulb until the mercury rises about three-fourths of the way up the stem. This will cause a suitable bulb to be blown in the soft glass at the top.

This plain rounded end is the easiest way of finishing the top of the thermometer. A ring top can be made by drawing out the stem to about 2 mm. diameter, forming the bulb while the thicker glass is still soft, cooling, and forming the ring afterwards. Another satisfactory top is formed by drawing the stem out and then melting back the smaller diameter rod to form a sphere. The thermometer can then be suspended by a wire fastened around the constricted neck.

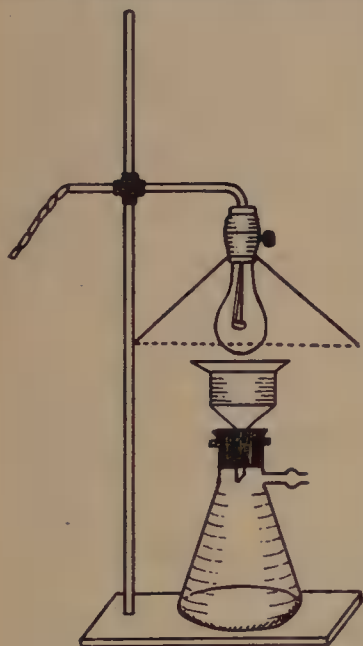
We invite contributions of articles for publication in "The Chemist Analyst," short original articles on methods of analysis or laboratory suggestions (not over 600 words). We will be glad to pay contributors for all articles that we can use.

Radiant Heat for Filtrations

LOUIS H. GOODSON

Maltbie Chemical Co., Newark, N. J.

A simple method of keeping a funnel and its contents hot during a filtration is illustrated in the accompanying sketch. The source of heat is an electric light bulb with a reflector. An ordinary reflector, originally designed for use in photographic lighting, proved to be satisfactory. Of course the size and type of bulb can be varied, but a clear 150 watt lamp worked nicely for heating 6" funnels as high as 80° C. For suction filtrations best results are obtained by wrapping the funnel with a cloth so that the filter plate and the lower portions of the funnel do not become cold. The amount of heat applied is conveniently regulated by raising and lowering the light bulb. A watch glass placed over the funnel serves to prevent excessive solvent losses. Some of the advantages of this set-up are: (1) accurate regulation of the temperature is easy; (2) the fire hazard created by the lamp bulb is not great; (3) costly jacketed funnels are not required as this set-up is easily adapted to nearly all sizes and types of funnels; (4) the necessary parts are usually available at low cost.



Removal of Carbon from Apparatus

C. W. BENNETT

Western Illinois State Teachers College, Macomb, Illinois

Often one is confronted by the necessity of cleaning a deposit of carbon out of a test tube or flask, etc., in which sugar or other organic compound has been heated. Of course this may be removed by the slow process of heating the glassware to redness and oxidizing the carbon. We have found that a little dry potassium chlorate, KClO_3 , added to the dry glassware and heated cautiously, will remove the carbon rapidly with little danger to the apparatus. Usually the carbon will ignite with a little "pop" and occasionally the tube will behave like a miniature Roman candle, but if care is exercised there should be no danger to the operator. After the reaction is complete, the glassware may be cooled and the potassium salts washed out with hot water.

Stirring Rod for Test Tubes

WRIGHT E. OWEN

Grinnell College, Grinnell, Iowa

In performing simple chemical tests many students are tempted to shake reaction mixtures containing dangerous chemicals by holding the thumb over the mouth of the test tube. If a gas is evolved in the reaction, corrosive chemicals may fly into the face or eyes when the thumb is released.

The use of stirring rods, shaped in the form shown in the illustration, is suggested. A vertical motion of this type of rod has proven to be quite satisfactory for mixing substances in test-tubes, especially in the hands of inexperienced students.

The rod should be about 7" long. It is fashioned from ordinary 5-6 mm. lime glass rod by softening one end and pressing it at a 45° angle against an asbestos or "Transite" board while the glass is still soft. The bent portion should be again softened and flattened as much as possible by pressing it against the board with the file or tongs.



FRONT



SIDE

The making of this simple and handy device provides an excellent exercise in glass manipulation.

An Aid to Rapid Weighing

LEWIS APPLETON

Allentown Testing Laboratory, Allentown, Penna.

In the iron and steel laboratory, where many samples have to be analyzed daily, the time consumed in weighing out definite amounts of chips or drillings can be shortened appreciably by using a steel-bladed spatula for the weighing of the samples. By slightly magnetizing the steel blade of the spatula, one or several particles of iron or steel can easily be held on the blade until the spatula is tapped gently with the forefinger. When the balance point is approached, the magnetized blade can be used to add or remove particles of the desired size, while the balance is still in motion, if care is exercised by the analyst to prevent the blade from touching the weighing scoop.

In this laboratory the initial amount of drillings is weighed out with the regular sized spatula, but the final adjustment is made more quickly with a magnetized nail, ground at the end to a fine point. The nail, 8 or 10 penny, is bent to about a 45° angle and inserted in a cork stopper of suitable size; another nail, not bent and not magnetized, is inserted in the other end of the stopper to serve as a handle. With this simple device the drillings in the weighing scoop can be conveniently and safely approached with the balance in motion, and the final adjustment is quickly made. After a little practice, it is possible to weigh out a sample in one minute, which represents a considerable saving of time over the usual method.

A small improvised electro-magnet has also been used satisfactorily for weighing out samples of magnetic material, using a "keycase" battery with single cell as the source of magnetizing current and a wire-wound nail as the magnet.

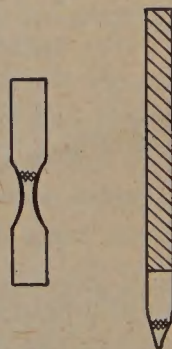
Auxiliary Valve for Laboratory Water Pumps

KARL E. RAPP

Kentucky Agricultural Experiment Station, Lexington, Ky.

Frequently laboratory aspirators operate at reduced efficiency and may cause filtrate contamination unless constantly watched. This is caused by failure of the pump valve during a variation of water pressure, whereupon tap water is drawn back into the suction system. Unfortunately this is true of a large percentage of new pumps as well as old ones. A very simple yet highly effective device is described in the following.

An auxiliary valve, to be inserted between the aspirator and suction system, is made. A short length of 8 mm. Pyrex tubing is constricted to approximately 3 mm. without drawing out. This provides a heavy wall at the constriction into which the needle valve plunger is to be ground. The plunger is made from a piece of 5 mm. glass rod about 8 cm. in length, one end of which has been pulled off to a point. The sleeve is set in a cork stopper held by a clamp. The plunger is coupled to the shaft of a small motor mounted above the sleeve. Grinding first with fine "Carborundum" and later with emery dust, a water tight seal is obtained. In the accompanying scale drawing the shaded portion of the rod shows the part cut off after the grinding has been completed. The cross-hatched areas indicate the ground seal. Finally the short plunger is put in place and both ends of the sleeve are fire polished, at the same time shrinking the longer end to trap the plunger.



This device allows no leak back, whether the water pressure is diminished gradually or suddenly.

· · MISCELLANY · ·

Preparation of Asbestos for Gooch Crucible Mats

GAYLORD M. VOLK

University of Florida, Agricultural Experiment Station, Gainesville, Fla.

Hoffman (*Chemist Analyst*, 31, 48) has recommended use of the Waring Blendor for preparing the suspension of asbestos fibers. Considerable trouble has been experienced by the writer in using such a preparation, as the short fibers thus obtained do not orient themselves horizontally to form a tight mat capable of retaining fine precipitates. Under certain conditions it may be advantageous to employ that high speed dispersion method. However, scarcity of high grade asbestos makes it advisable to proceed with caution lest large quantities of it be rendered unfit for use in many cases.

Removal of Frozen Glass Stoppers

W. M. REID

Monmouth College, Monmouth, Ill.

Numerous glass stopper jacks have been invented for the removal of frozen stoppers. Various methods of tapping or heating bottles to remove stoppers have also been described. A drawback to the jacks is their unavailability when needed, while the latter methods are attended with more or less risk of breaking the bottle.

An extremely simple method which has neither drawback is merely to invert the bottle. Within a few minutes the stopper will usually loosen as the liquid penetrates between the stopper and the neck of the bottle. If the bottle contains a viscous liquid or if the stopper is corroded, it may be necessary to invert the bottle in a beaker of water so that liquid will penetrate from both directions. The progress of the liquid may be watched and the stopper removed as soon as penetration is almost complete. Even a bottle which has been corroded for years may be opened after being inverted for a day or two in a beaker of water.



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